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#### Research Paper

## Water-responsive rapid recovery of natural cellular material



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#### ABSTRACT

Insight into the stimuli–responsive behaviour of biological materials with hierarchical microstructures is essential for designing new sustainable materials and structures. Shape memory, self-healing and self-repairing will become valuable characteristics of advanced materials. Here we report the water-triggered shape recovery of a natural biological material, the luffa sponge. The longitudinally crushed luffa sponge column can recover up to 98% of its original shape after it is immersed in water. The mechanical properties of the luffa sponge can also be recovered, to a large extent, after a subsequent drying process. The effects of strain rate, crushing strains, loading cycles, and temperature/duration of water treatment of the drying process on the shape recovery ratio and the energy dissipation recovery ratio have been investigated. The results from this study have demonstrated that the luffa sponge material possesses remarkable shape memory effects and mechanical recovery features which could be exploited or biomimicked for the design of water-responsive smart materials undergoing large deformations.

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#### 1. Introduction

Cellular materials with hierarchical microstructures have attracted much attention due to their excellent mechanical performance and the potential to achieve multi-functions such as vibration and shock isolation, thermal insulation, catalyst support, and acoustic absorption. The ability to maintain structural integrity upon large deformation is essential to the reliability of these materials in diverse applications. Materials with hierarchical microstructures are susceptible to fracture under

large deformation. Stimulus–responsive and maintenance-free self-healing features may significantly improve the reliability of these materials. Biological organisms, especially various plants, utilize complex hierarchical micro-vascular networks which support a number of critical functions, such as temperature control, healing by tissue regeneration, waste removal, and nutrient transport and distribution. As a result of the selective evolution of living organisms over millions of years to construct composite materials, the branching and size of these microvascular networks have been optimized or partially optimized to

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minimize the energy required for fluid circulation. In this way, the biological systems can fulfil the demands of the natural environment using a rather limited chemical repertoire. The cellulose microfibers are the major base material for the cell wall of the microvasculature of plants (Gibson, 2012). It is envisaged that the microvasculature of plants may possess Shape Memory Effect (SME), self-healing and self-repairing features. At the same time, various materials and structures employ the microvascular system to achieve self-healing function (Ahmad and Thambiratnam, 2009; Shen et al., 2013). A recent discovery of the ability of Cellulose Nano-Whiskers (CNWs) to act as watersensitive switches provides a new strategy to combine different types of shape memory switches into one material by composite methods (Luo et al., 2011; Zhu et al., 2012). This finding expands the approaches to tailoring the SME of the nano-composites and to developing new shape memory polymeric composites for various potential applications (Hu et al., 2012).

The fruits of Luffa Cylindrica (LC) have a netting-like fibrous vascular system. When they are dried, the fibrous network forms an open cell cellular material. In a recent study (Shen et al., 2012), we discovered that the luffa sponge material exhibits remarkable stiffness, strength and energy absorption capacities that are comparable to those of some metallic cellular materials in a similar density range. The luffa sponge also has recycling capability and triggered biodegradability (John and Thomas, 2008; Oboh and Aluyor, 2009). The importance of biological materials such as the luffa sponge is growing as we search for sustainable solutions using new or unexploited materials.

Only a limited amount of research has been conducted on the luffa sponge. The previous research focused on using the luffa sponge as a source of bio-fibres and bio-composites. Those investigations indicated that the luffa sponge could be a potential alternative material for packaging (Mazali and Alves, 2005), water absorption (Bal and Bal, 2004; Demir et al., 2006), and waste water treatment (Laidani et al., 2011; Oboh et al., 2011). The luffa fibres were also used as the reinforcement fibre for other materials (Boynard and D'Almeida, 2000; Ghali et al., 2009; Laranjeira et al., 2006; Paglicawan et al., 2005; Tanobe et al., 2005) and cell immobilization for biotechnology (Chen and Lin, 2005; Roble et al., 2002; Tavares et al., 2008; Zampieri et al., 2006). Oil can be extracted from luffa seeds for industrial use (Bal and Bal, 2004). The oil extracted from LC is finding increasing use in the production of biodiesel which is now gaining wide acceptance because of low CO2 emission and other considerations (Ajiwe et al., 2005).

Here we report the water-driven recovery properties of the luffa sponge material and its component microstructures-luffa fibres. The major influential parameters for the shape recovery ratio and the energy absorption recovery ratio at macroscopic level are identified. Several possible mechanisms behind the physical recovery process are suggested and discussed.

#### 2. Materials and methods

#### 2.1. Luffa sponge materials and specimens

The luffa sponge used in our experiments was obtained from pharmacies in Australia which was sold as a bath sponge. A brief description about the treatment procedure for manufacturing these bath sponges from natural luffa fruits was provided by the supplier. The luffa fruits were harvested after they were fully mature with their skin turning brown as shown in Fig. 1a. The dried luffa fruits were slightly squashed laterally to crack and remove the skin. Then the two ends of luffa fruits were cut and the seeds were removed. The original luffa sponges were bleached using liquid chlorine bleach (4%) for about 1 h to improve their appearance by making them whiter. After that, they were soaked in clean water for half an hour and then dried in the sun. We conducted comparative experiments with results showing that both bleaching and laterally squashing would only reduce the strength of the natural luffa sponge material slightly (around 5%). The chemical composition of the luffa sponge depends on several factors, such as the plant species, weather condition, soil, etc. A set of reference values for the chemical composition of the luffa sponge can be found from a previous study (Siqueira et al., 2010).

Cylindrical specimens were used for all the compression recovery tests for luffa sponge columns as shown in Fig. 1a. The cross-sections of these specimens are shown in Fig. 1b. The morphological features of the luffa sponge column along the longitudinal direction are shown in Fig. 1c. As the component microstructure of luffa sponge material, the SME of luffa fibres was also investigated phenomenally through bending and laterally compression. For the bending recovery tests, luffa fibres of arbitrary cross-section with branches as shown in Fig. 2a were used. Different connection patterns of luffa fibres to form the 3D luffa sponge column are illustrated in Fig. 2b. Luffa fibres with an approximately circular cross-section were used for recovery tests on micro-tunnel structures of luffa fibres through lateral compression as shown in Fig. 2c.

Two luffa sponge columns (approximately 50 mm high) were cut from a single luffa sponge using a bandsaw cutting machine as comparison counterparts. After the initial cutting, the specimens were milled further to make the two end surfaces smooth and parallel. For calculation purpose, the cross-sectional area was taken as the enclosed section (ignoring any internal voids). The measured maximum diameter of the dry luffa sponge specimens tested was within the range of 55-86 mm. According to the previous extensive research (Ashby et al., 2000) on other cellular materials, the specimen size effect would be negligible for foams when the dimension of specimen is sufficiently larger than the cell size, i.e., 7 times of the cell size for metallic foams. Thus the specimen for the luffa sponge should be large enough to eliminate the specimen size effect. The maximum available size along the radial direction for the luffa sponge is from the whole section of the luffa sponge column. For most of the luffa sponge columns, the cross-sectional areas at two ends of the specimen are different. Thus two thin layers of luffa sponge slices (approximately 5 mm thick) were cut at the two ends to measure the cross-sectional area for both ends (Shen et al., 2012).

Because the cross-section for most specimens was not perfect circular, the actual cross-sectional area was determined from photographs taken of each measurement slice and processed using the image processing software Photoshop. The actual cross-sectional areas from the two measurement slices were then averaged. An equivalent diameter was calculated from the

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